MISSOURI DEPARTMENT OF TRANSPORTATION
MATERIALS ENGINEERING
Jefferson City, Missouri

Test Method
MoDOT T34
DETERMINATION OF PHOSPHORUS IN LUBRICANTS

1.0 SCOPE

This method describes a procedure for determining the percent phosphorus in lubricants.

2.0 REAGENTS AND APPARATUS

(a) Nickel crucible about 25 ml capacity.

(b) Ammonium Molybdate solution. To an 800 ml beaker add 600H_2O, 65 g $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, 225 g NH_4NO_3 and 15 ml NH_4OH, all reagent grade. Heat gently until the crystals have dissolved, filter through Whatman No. 41 paper, and dilute to 1 liter.

(c) Phenolphthalein indicator solution. Dissolve 1 g phenolphthalein in 100 ml of 95% C_2H_5OH.

(d) 0.15 N Sodium Hydroxide solution. Dissolve 6 g of Reagent Grade NaOH in H_2O and dilute to 1 liter.

(e) Potassium Permanganate solution. Dissolve 25 g of Reagent Grade $KMnO_4$ in H_2O and dilute to 1 liter.

(f) 0.15 N Nitric Acid solution. Dilute 9.5 ml of Reagent Grade HNO_3 to 1 liter. Standardize against Reagent Grade NaOH or Na_2CO_3. The phosphorus equivalent of the solution is equal to 0.00135 x Normality.

(g) KNO_3 wash solution. Dissolve 11 g Reagent Grade KNO_3 in H_2O and dilute to 1 liter.

3.0 PROCEDURE

If the phosphorus content of the material is less than 0.07%, weigh a 5 g sample; if more than 0.07% weigh a 1 g sample. Weigh the proper size sample to the nearest 0.1 mg and

transfer to a nickel crucible. Add 0.5 g each of Na_2O_2 and ZnO, both Reagent Grade. Add the same reagents to another nickel crucible and carry through the test as a blank. Place the crucible on a hot plate and maintain at 90-110 C for 30 min. Remove from the hot plate and allow to cool. Add 1 ml of benzene and ignite. Continue burning until all the oil is consumed, warming on the hot plate again if necessary. After the oil is completely burned, heat over a meeker burner until most of the carbon is oxidized and the mass begins to melt. Do not allow the mass to fuse completely.

Allow the crucible to cool and wash as much of the contents as possible into a 250 ml beaker. Rinse the crucible into the beaker several times with 1-1 HNO_3 and hot H_2O. The final volume should be about 150 ml, and should include 20 ml of concentrated HNO_3. Filter through a No. 41 Whatman paper into a 500 ml flask. Heat the filtrate to boiling and add $KMnO_4$ solution, drop by drop, until the solution retains the purple color. Destroy the excess KMnO4 with a few drops of 3% H_2O_2.

Cool the solution to 37-45C and add 50 ml of the ammonium molybdate solution. Stopper the flask, shake vigorously for 3-5 min, and allow the precipitate to settle for about 1 hr. Filter through No. 42 Whatman paper and wash well with the KNO_3 wash solution.

Place the filter paper and precipitate in the original flask and add 15 ml H_2O and several drops of the phenolphtalein indicator solution. Pipette 10 ml of the 0.15 N NaOH solution into the flask and agitate to dissolve the precipitate. The solution should be alkaline at this point; if not, pipette another 10 ml NaOH into the flask. Add the same total amount of NaOH solution to the blank. Dilute to 150 ml with H_2O and titrate the excess NaOH with 0.15 N HNO_3.

4.0 CALCULATIONS

Calculate the percent phosphorus in the sample as follows:

$$\% P = \frac{(B - S) \times E}{\text{Wt. of sample}} \times 100$$

Where:

B = ml HNO_3 to titrate the blank

S = ml HNO_3 to titrate the sample

E = Phosphorus equivalent of the HNO_3 solution.

Report as: % Phosphorus (P), to the nearest 0.01%